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Phase Transitions and Amorphization of CaWO₄ at High Pressure

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Beamline(s): X17C

Introduction: The phase transitions in CaWO₄ were studied by in situ x-ray diffraction at high pressure up to 65 GPa. The previously noted phase transition was observed at 12 GPa and the high-pressure phase was by the first time characterized. We found also that CaWO₄ becomes amorphous at pressure exceeding 40 GPa. A new high-pressure phase was determined at around 40 GPa and 477 K.

Methods and Materials: The experiments were performed using a collimated x-ray white beam of 20 micron by 20 micron in a diamond anvil cell at a diffraction angle $2\theta = 13^\circ$. CaWO₄ powder of pure quality (97.78%) was placed along with a gold pressure standard in a 100-micron hole in a 301 SS gasket. CaWO₄ acts as its own pressure medium. The pressure-induced transformations were monitored in three different samples.

Results: We performed energy-dispersive synchrotron powder diffraction of CaWO₄ to pressures an order of magnitude greater than achieved in earlier studies [1]. The phase transition previously determined by Raman spectroscopy [2] was observed at 12 GPa and the high-pressure phase characterized as a wolframite-type structure (SG: P2/c, No. 13). On pressure release CaWO₄ reverts to its ambient pressure scheelite structure (SG: I41/a, No. 88). The wolframite phase remains stable up to 40 GPa. However under further compression the diffraction peaks disappear and some pronounce broad scattering, commonly observed in amorphous solids, is present. Clear peaks were not recognized after pressure releasing, but by heating to 477 K after 2 hours a pattern corresponding to the ambient pressure phase is obtained. Finally after heating to 477 K and quenching an amorphized sample at 45 GPa new diffraction lines were observed indicating the existence of a new high-pressure phase stable at high temperature. This phase is found to be monoclinic (SG: C2/m, No. 12). The volume decrease in going from the room temperature phase to this phase is estimated to be 13.2%. Similar phase have been observed in other tungstates showing a volume collapse of about 12%, which suggests that the existence of a high-pressure and high-temperature phase could be a common behavior of all tungstates. The occurrence of this new high-pressure high-temperature phase gives additional support to the idea that amorphization takes place because of the frustration of a phase transition, which in our case was kinetically favored by the heating.

Conclusions: This study deepens the knowledge of the behavior of CaWO₄ under pressure and provides information that linked to the performance of electronic structure calculations under pressure may help to improve the microscopic understanding of the phase behavior of CaWO₄ and other tungstates.

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References:

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